

AFRL-ML-TY-TP-2000-4551



THE APPLICATION OF FT-IR SPECTROSCOPY TO TURBINE ENGINE EXHAUST MEASUREMENT

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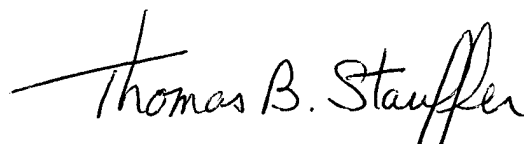
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ABSTRACT

An advanced multiple gas analyzer based on Fourier transform infrared (FT-IR) spectroscopy has been used to successfully measure the exhaust composition from a gas turbine engine combustor rig at the Arnold Engineering Development Center (AEDC), Arnold Air Force Base (TN). The advanced FT-IR multigas analyzer was able to demonstrate its ability to not only measure the species traditionally measured at the facility (water, CO₂, CO, NO, NO₂, and total hydrocarbons), but also to measure many other species, including sulfur dioxide, formaldehyde, ethylene, propylene, methanol, formic acid, and nitrous acid. A side by side comparison of the single gas analyzers currently employed by AEDC and the FT-IR Multigas analyzer showed good agreement. The capability of a single relatively low-cost, small sized Multigas analyzer to measure most of the important products of combustion makes it an attractive alternative to existing emissions measurement systems. The Multigas analyzer may be used to measure the exhaust gas composition of aircraft engine combustors that are regulated by the FAA as well as stationary source engines that are regulated by the EPA.

INTRODUCTION

Under a DOD sponsored SBIR Phase II Program, Advanced Fuel Research (AFR) has been exploring the application of Fourier transform infrared (FT-IR) spectroscopy for gas analysis in turbine engine exhaust monitoring.¹ The importance of collaborating with Sverdrup Technology, Inc. at AEDC during this exploration is obvious. AEDC's Engine Test Facility (ETF) is used routinely for testing combustor and full propulsion systems including stationary power sources and turbojet, turbofan, and ramjet air breathing engines. Gas phase emissions testing before flight is one key responsibility of Sverdrup Technology at AEDC.²

Traditionally, engine emissions are analyzed for species such as O₂, water, CO₂, CO, total hydrocarbons, NO and NO_x using an array of single gas analyzers (SGA). This large number of individual analyzers results in both a high initial cost, as well as a significant investment in maintenance overhead and calibration gas infrastructure. In addition, each instrument has a limited dynamic range and must be calibrated on an hourly basis using unique reference gases. Lastly, if analysis for a new species is desired, a new instrument must be purchased, if it is even available.

FT-IR gas analysis, on the other hand, is a full spectroscopic technique that makes it possible to monitor many species simultaneously in a single instrument. FT-IR is based on the fact that every molecule, except homonuclear diatomics, has a unique set of rotational and vibrational frequencies that absorb and emit infrared energy in a characteristic manner. In general, it is possible to identify and quantify gases based on the location and magnitude of these absorptions, which occur throughout much of the infrared. Since a FT-IR spectrometer is capable of recording infrared absorptions from about 500 cm⁻¹ (20 μm) to 8000 cm⁻¹ (1.3 μm), it can be used to easily identify and quantify an almost unlimited variety of compounds.³ **Figure 1** plots a composite infrared spectrum indicating many of the different compounds detectable in gas turbine exhaust using FT-IR, while **Table I** provides more detail. As can be seen, a wide range of compounds of interest can be detected with good sensitivity using FT-IR, even in the presence of high water concentrations (up to 50% has been investigated at AFR). This makes FT-IR unique in its ability to monitor many species simultaneously over a very wide concentration range, from sub-ppm to percent. In addition, the ability to measure on a wet gas stream makes it possible to measure the exhaust with minimal sample conditioning. As can be seen, a single FT-IR based gas analyzer is capable of consolidating several racks of single gas analyzers into a single compact instrument, with the added benefit of

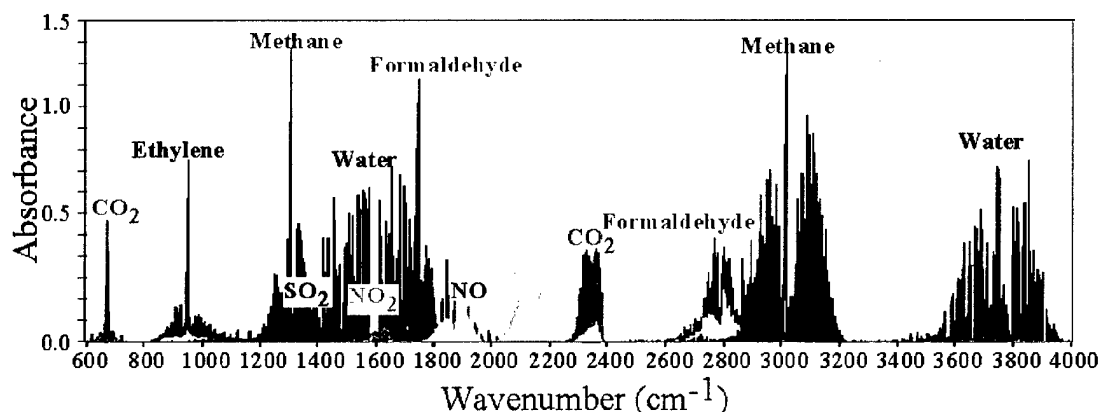


Figure 1. Composite infrared spectrum indicating many of the compounds found in turbine exhaust that can be detected using FT-IR. The concentrations of CO₂ and water have been suppressed in order to simplify viewing of the trace species.

Species	Calibrated Range	Detection Limit (ppm)	Analysis Region (cm ⁻¹)	Uncertainty Estimate (% Reading)
H ₂ O	0 to 50%	85	1097-1169	±4.0%
CO ₂	0 to 20%	25	2211-2236	±2.5%
CO	0 to 200000 ppm	0.11	2164-2185	±2.5%
NO	0 to 2500 ppm	0.51	1849-1858	±2.5%
NO ₂	0 to 410 ppm	0.19	1584-1589	±2.5%
NH ₃	0 to 10000 ppm	0.06	907-975	±2.5%
CH ₄	0 to 50000 ppm	0.77	2839-2922	±2.5%
Formaldehyde	0 to 360 ppm	0.13	2672-2839	±2.5%
Ethylene	0 to 500 ppm	0.10	900-1000	±2.5%
Methanol	0 to 520 ppm	0.12	964-1090	±2.5%
SO ₂	0 to 2000 ppm	1.20	1100-1173	±2.5%
Propylene*	0 to 110 ppm	0.22	901-1020	±15.0%
Propane*	0 to 100 ppm	0.80	2839-2921	±15.0%
Jet Fuel (A)	0 to 2200 ppm	2.00	2790-2922	±15.0%
Formic Acid	0 to 15 ppm	0.11	1047-1148	±10.0%
Nitrous Acid*	0 to 2 ppm	0.03	823-889	±15.0%

Table I. Species currently measured by the FT-IR gas analyzer that are of interest for combustion monitoring. The detection limits for the Multigas are estimated for 0.5 cm⁻¹ resolution and a 10 second scan time with the gas cell held at 150°C (to prevent condensation of the wet gas stream). This limit is based on 3x the Classical Least Squares (CLS) reported concentration for a zero concentration sample, and is the detection limit using the spectral region indicated. If other spectral analysis regions are used, the detection limits can change. The analysis regions were selected to reduce the impact of water interference, so water will not affect these limits. Several species have the same or overlapping analysis regions, which is possible because of the CLS analysis routine used. The column labeled "Calibrated Range" indicates the range of concentrations for which references have been obtained, but does not represent the maximum range measurable by the instrument. Included in the table is an estimate of the total measurement uncertainty for each species, and incorporates uncertainties in the calibration source (±2% for calibration gases and permeation tubes, and up to ±15% for a liquid source), dilution mass flow controllers (±1% each), and pressure transducer uncertainty (±0.7%). These references obtained from: "Gas Analysis Manual for Analytical Chemists, Vol. 2: Quantitative Reference Spectra for Gas Analysis," Infrared Analysis, Inc., Anaheim, CA.

Species	Calibrated Range	Make	Model	Method	Calibration Gas Composition	Uncertainty Estimate (% Reading)
CO ₂	0-5%	CAI	ZRH-2	NDIR	4.15%	±2.1%
	0-20%				4.15%	±2.3%
CO	0-1000 ppm	CAI	ZRH-2	NDIR	803 ppm	±2.3%
NO	0-25 ppm	Thermo Electron Instruments	10	Chemi-luminescence	196.2 ppm NO _x	±4.3%
	0-250 ppm				196.2 ppm NO _x	
NO _x	0-25 ppm	Thermo Electron Instruments	10	Chemi-luminescence	196.2 ppm NO _x	±4.0%
	0-250 ppm				196.2 ppm NO _x	
THC	0-30000ppm C ₁	Siemens	Fidamat 5E-A	FID	7020 ppm C ₃ H ₈	±3.9%
O ₂	0-25%	CAI	ZAJ	Paramagnetic	20.8%	±1.3%
Dew Point	-40-120°F	EG&G	300	Chilled Mirror	n/a	±0.36°F

Table II Single gas analyzers used by AEDC. Included in the table is an estimate of the total measurement uncertainty for each species, and incorporates the inherent instrument accuracy, repeatability, and calibration gas uncertainty.

providing concentrations on species previously considered difficult to measure, such as formaldehyde and ammonia. Another benefit of FT-IR is its ability to perform data validation, since the infrared spectrum recorded is stored along with the concentration report. This means that any unusual data points can be re-evaluated or validated long after the test was completed. No such validation is possible with a single gas analyzer.

Calibration of a FT-IR gas analyzer requires the generation of a spectral library containing all the compounds of interest spanning the range of expected concentrations. When analysis for a new compound is desired, it is a simple matter of measuring a new set of references for that species. Assuming the analyzer is well designed, there is no need to record these references for each individual instrument manufactured (since the infrared spectrum for a given molecule is a fundamental property). In fact, all of the calibration spectra used for this project, including the percent level compounds such as water and CO₂, were measured on a different instrument and gas cell than was used to take the data. In order to confirm the correct operation of the FT-IR analyzer on a day to day basis, a single calibration gas can be measured to verify proper operation of the instrument. This gas could be as simple as dry nitrogen and ambient air, although typically a calibrated gas mixture such as CO or ethylene in nitrogen is used. This simplified calibration check significantly reduces the overhead necessary to support the analyzer.

AFR chose the On-Line Technologies, Inc. (On-Line) FT-IR based Multigas analysis system (MG-2010) as the core component for this study. The system uses proprietary, aberration corrected long-path gas cells (series 20/20™) integrated with an advanced, high-speed FT-IR spectrometer designed for industrial applications. The gas cell and spectrometer are integrated into a single, compact instrument (20"wide x 14"tall x 30"deep), making the MG-2010 an ideal candidate for continuous emission monitoring (CEM) applications. In order to maximize signal to noise and photometric accuracy, the mercury-cadmium-telluride (MCT) detector is digitally linearized. This advanced feature, along with the careful optical design, results in a system with almost no maintenance, excellent stability, and minimal instrument to instrument variability (1%-2%, maximum). As mentioned above, this low variability between instruments allows a single spectral database to be used for any On-Line instrument, so the high cost of individual calibrations is eliminated.

The MG-2010 is capable of simultaneously measuring gas concentrations for a wide range of compounds with detection limits and accuracy comparable to single gas analyzers (See **Tables I** and **II**). In addition, this instrument is capable of making many measurements that cannot easily be obtained in real time by any other commercial method (i.e. formaldehyde or ammonia), which makes FT-IR gas analysis one of the most versatile and cost effective instruments for emissions monitoring on the market today.

In order to evaluate the performance of the MG-2010 analyzer compared to a set of single gas analyzers used for engine emissions monitoring, AFR provided AEDC with a Multigas analyzer for a two-month evaluation. During this time, the MG-2010 collected data in parallel with the array of single gas analyzers used on one of AEDC's combustion test rigs (see **Table II**). This test rig is capable of controlling fuel flow rate to combustion zones to 50 lbm/sec, inlet air pressure, temperature, and mass flow rate, as well as combustor exit pressure. The facility is also equipped to measure both static and total pressure throughout the flow path, and dynamic pressures for analysis of instabilities in the combustion process. Exhaust gas analysis is performed using an array of gas analyzers, including CO₂, dew point temperature, CO, NO, NO_x, total hydrocarbons (THC), and O₂. The emissions system complies with the SAE aerospace recommended practice for analysis of aircraft engine exhaust gas (ARP 1256) and EPA requirements for stationary source emissions measurements.

The MG-2010 FT-IR Multigas analyzer (Multigas) used at AEDC incorporates a stock On-Line series 20/20™ gas cell, which provides a folded optical path of 5.6 meters with a sample volume of 1600 cm³. The aberration correcting optics in this gas cell design results in an optical throughput of more than 60% at 150°C. Recently, AFR has developed a low volume (200 cm³), 5.11 meter gas cell based on the 20/20™ design.¹ This cell has almost an order of magnitude faster time response than the original cell (4 seconds vs. 35 seconds at 10 SLPM) with comparable optical throughput (~50% at 150°C). The 20/20™ gas cell design is ideal for fieldwork and industrial applications because the multipass mirrors require no alignment and are easily field swappable. The 5-meter pathlength of these gas cells was chosen as a compromise between good sensitivity and tolerable water interference on compounds such as NO, NO₂ and SO₂. Analysis of the gas species concentrations is performed using a classical least squares (CLS) routine that compares the spectra recorded from the combustor exhaust to a standard library of calibrated reference spectra (±2.5% typical uncertainty), as mentioned above. Most references are recorded at multiple concentrations in order to account for any non-linearity of the absorbance with concentration. This non-linearity is most pronounced for small molecules with narrow rotational lineshapes, such as CO, and is caused by the 0.5 cm⁻¹ resolution of the spectrometer not fully resolving the true lineshape (~0.1 cm⁻¹ at one atmosphere). These non-linearities, as well as species crosstalk, are minimized by using analysis software that is designed to automatically select the reference spectrum that is closest to the concentration found in the sample being analyzed.

The analysis regions listed in **Table I** were selected to minimize any errors in the measurement due to water interference. This is most critical for NO₂, NO, and SO₂, which had less than 0.5 ppm error related to water interferences at the highest water concentration ranges encountered during the test (12%-13%). Additionally, each of the species listed on the table may interfere with the analysis of one or more of the other species, and this crosstalk is automatically taken into account by the software. The ability to cope with spectral overlap is a major advantage for FT-IR gas analysis, since the sample need not be dried or conditioned in any significant manner prior to measurement.

EXPERIMENTAL SETUP

The setup used to perform the FT-IR gas measurements is depicted in **Figure 2**. The exhaust gas is extracted from the test rig using the same sampling rake and heated transfer line used by the traditional AEDC analyzers. The sample is split between the single gas analyzer rack and the Multigas, so both systems are working with identical samples. Prior to entering the single gas analyzer rack, the sample stream, except for the total hydrocarbon sample, is dried using a membrane gas drier and filtered for particles. In order to account for the residual moisture in the sample stream, a dewpoint meter is used to measure moisture levels after the drier. Once analyzed, the measured CO₂, CO, and total hydrocarbon levels are used to back calculate the water concentration in the exhaust stream given the combustor stoichiometry, inlet air humidity, and fuel composition. This added level of complexity is required because the single gas analyzers will not work on high moisture samples.

As shown in **Figure 2**, the MG-2010 operates with minimal sample conditioning. The only conditioning required is particle removal, which is accomplished by passing the sample through two heated (150°C) particle filters (99.99998% combined efficiency for particulates larger than 0.6 μm). Particle filtration of the gas stream is essential when multipass cells are used, since losses in mirror reflectivity can significantly degrade sensitivity and shorten the maintenance interval. In order to automatically account for ambient pressure fluctuation as well as backpressure on the exhaust line, an absolute pressure transducer (±0.7%) is located in the exhaust line just after the gas cell. The analyzer automatically records this pressure and corrects the sample for number density. This correction is essential, since atmospheric pressure can vary widely, even at sea level (±5% is not uncommon). Single gas analyzers do not usually account for this, since the analyzer is calibrated often. A nominal flow rate of 10 SLPM was used to provide relatively fast time response, about 35 seconds for a 99% cell volume exchange.

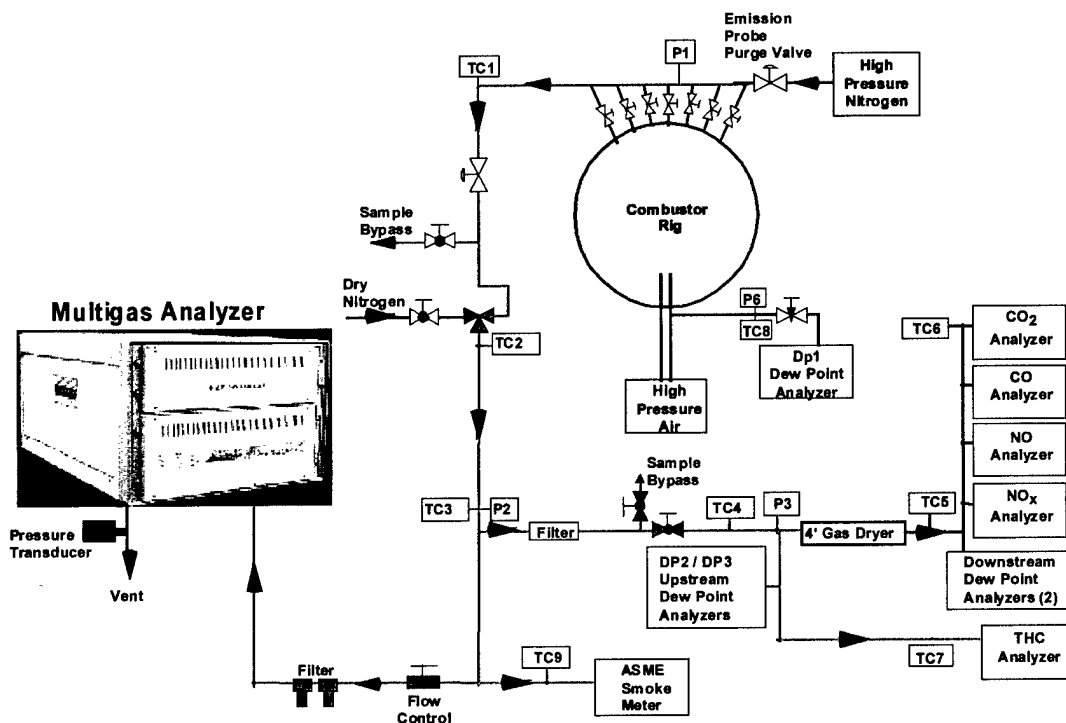


Figure 2. Experimental setup used to perform the head to head comparison of the Multigas to the single gas analyzers (SGA's). In this figure, TC# corresponds to thermocouple measurement points and P# are the pressure measurement points. Not all thermocouple and pressure sample points are shown. In addition, none of the zero or span gas lines are shown feeding the analyzers in order to simplify the drawing. Note that all the SGA's, excluding the THC analyzer, operate on a dried sample stream, while the Multigas performs measurements on the wet sample stream. The filters shown are heated to prevent condensation.

In order to validate the performance of the Multigas analyzer, calibration gases for CO (803 ppm CO, $\pm 2\%$) and NO (19.3 ppm, $\pm 2\%$) were used periodically over the course of the two-month evaluation. During these calibration checks, the CLS analysis routine calculated an average concentration of 790 ppm for CO and 19.7 ppm for NO, which are well within the combined uncertainties of the calibration gas ($\pm 2\%$) and analyzer ($\pm 2.5\%$).

RESULTS

A total of nine separate days of testing was performed on the test rig, covering a total of 123 test points. During these tests, the combustor was operated on either natural gas or a natural gas/steam combination. For a given test point, the combustor stoichiometry, flow, pressure and temperature are adjusted, and the rig is allowed to stabilize before a test point is acquired. It should be noted that many of the test points investigated were selected to map out the operational limits of the combustor, and were not intended to reflect its optimal operation. Prior to the start of each days test

program, a background reference free of contaminants was recorded on the Multigas to "zero" the instrument. This was done by purging the gas cell with dry nitrogen and co-adding a total of 200 scans (~2 minutes). During the test, the analyzer was operated continuously, collecting data at 20 second intervals (~32 scan average) with a nominal flow rate of 10 SLPM through the gas cell.

In order to compare the measurements obtained by the MG-2010 and the single gas analyzers (SGA), the SGA measurements were converted to a wet basis and correlation plots were generated for CO₂, CO, NO, NO_x, and total hydrocarbons (See Figures 3-7). As can be seen, good linear relationships exist for all the data. It should be noted again that the spectral references used to quantify these species were acquired on a different Multigas analyzer using a different gas cell (the 5.11 meter low volume cell was used for the references, while the 5.6 meter cell was used for the data collection).

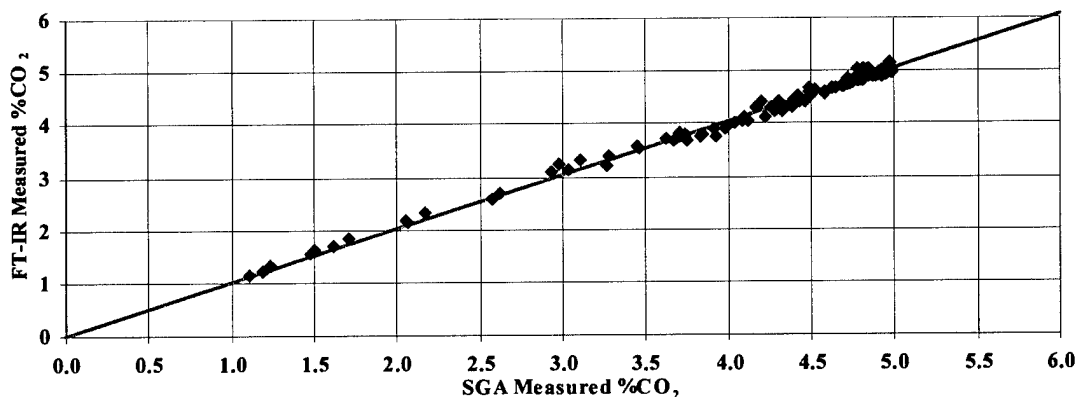


Figure 3. Correlation plot for CO₂. The best linear fit shown has a slope of 1.01 and zero offset. See text for details.

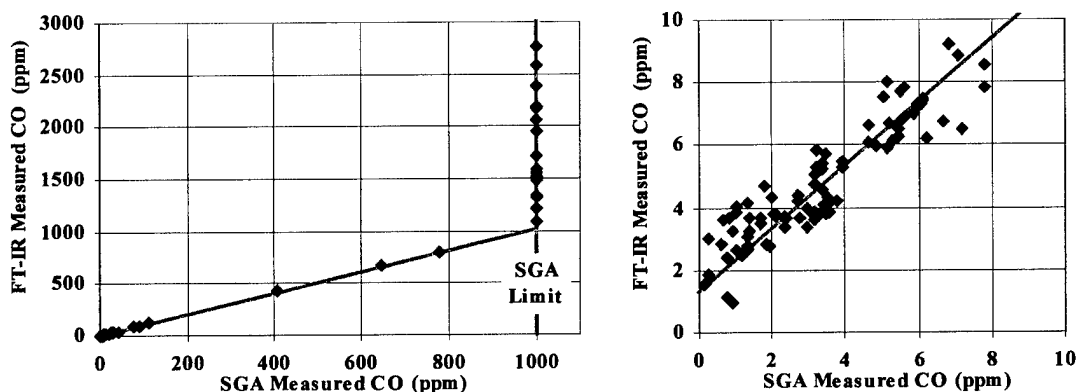


Figure 4. Correlation plot for CO. The graph on the left covers the entire dynamic range of the data, while the right expands the data from 0-10 ppm. The best linear fit shown has a slope of 1.01 and a +1.3 ppm offset. Note the SGA upper detection limit is 1000 ppm. See text for details.

CO₂ Comparison

Early on in this work, the CO₂ was analyzed using a band centered around 1000 cm⁻¹. This band originates from a vibrationally excited state, so it was extremely temperature dependent (~5% error in the analysis for a 5°C change in temperature). Once the analysis was moved to the 2200 cm⁻¹ region, which originates from a ground state vibrational level, excellent agreement between the SGA and the Multigas was observed (~1% high, see Figure 3).

CO Comparison

Figure 4 plots the correlation for CO. The SGA used at AEDC was limited to concentrations below 1000 ppm, but about 15% of the test points had levels above this. The Multigas has been calibrated to operate from sub-ppm up to 20% CO, and was able to provide quantitative results well above the SGA cutoff. The vast majority of data obtained (84%) is below 150 ppm, and in this regime the multigas was consistently 1% high

when compared to the SGA results. Between 150 ppm and 1000 ppm there are only three data points, and these points analyzed about 3% high. The lack of data in this middle region makes it difficult to determine a true uncertainty, although the 803 ppm CO calibration gas (±2%) mentioned above analyzed low by 1.5% in the Multigas. Lastly, the CO analysis on the Multigas appears to have a +1.3 ppm offset when compared to the CO analyzer. This offset highlights one of the advantages of FT-IR gas analysis: post-test validation, which is something that SGA's cannot provide. Upon re-examination of the FT-IR spectral data for test points reported to have near zero concentration by the SGA, the level of CO reported by the Multigas analysis program was verified to be correct. The discrepancy is thought to be due to one of two sources: SGA zero drift or incomplete FT-IR gas cell volume exchange. AEDC has indicated that the CO analyzer used during the tests did have a slight zero drift problem, which could easily account for the observed mismatch near zero ppm.

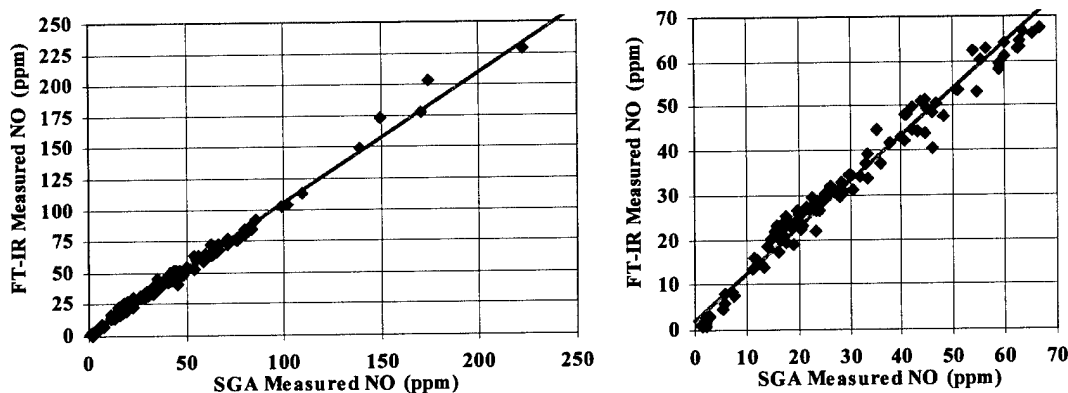


Figure 5. Correlation plot for NO. The graph on the left covers the entire dynamic range of the data, while the right expands the data from 0-70 ppm. The best linear fit shown has a slope of 1.04 and a +2 ppm offset. See text for details.

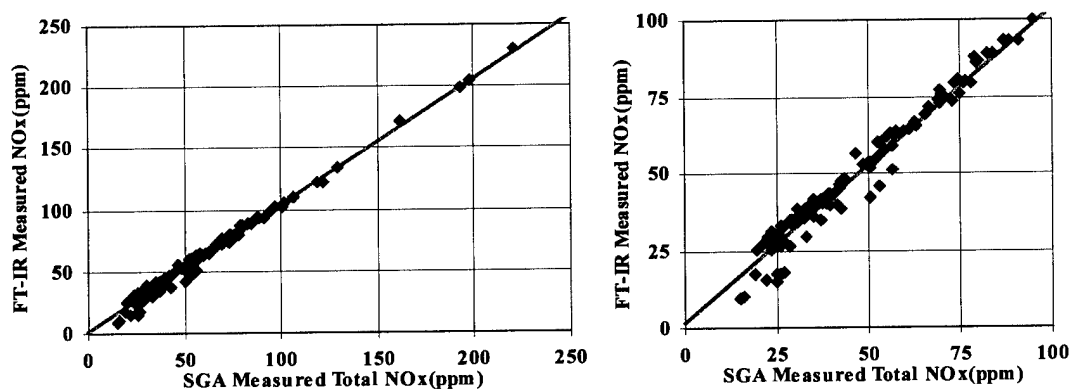


Figure 6. Correlation plot for NO_x. The graph on the left covers the entire dynamic range of the data, while the right expands the data from 0-100 ppm. The best linear fit shown has a slope of 1.02 and a +2 ppm offset. See text for details.

Residual exhaust gas from previous test points could also account for this mismatch, since some test point series are taken in rapid succession. The new 5.11 meter, 200 cm³ gas cell developed for the Multigas reduces the analyzer response from 35 seconds to 4 seconds (at 10 SLPM), and will eliminate the cell volume exchange issue from the equation.

NO and NO₂ Comparison

Figures 5 and 6 plot the correlation between the analyzers for NO and NO_x. As can be seen in Figure 5, the Multigas NO data is consistently 4% high when compared to the SGA results. Although this is well within the combined uncertainties of the two measurement techniques (~6.7%), experience at AFR has shown that sample drying systems have a tendency to shift NO₂ to NO. This was seen in the present data as well. Fortunately, the total NO_x appears to be largely conserved, and is usually the best way to compare the

Multigas performance for NO_x. In Figure 6, the total NO_x measured with the Multigas (NO+NO₂) reads approximately 2% high when compared to the SGA results, which is still well within the uncertainty of the two measurements.

A third nitrogen containing compound was detected in the exhaust stream: nitrous acid (HNO₂). This compound correlates best with NO₂ and ranged from 0.03 to 1.3 ppm. On average, HNO₂ was found at about 2.5% of the NO₂ concentration with a -0.06 ppm offset.

Total Hydrocarbon Comparison

The last direct correlation between the Multigas and the single gas analyzers is shown in Figure 7. In this plot, total hydrocarbons are measured by AEDC using a flame ionization detector (FID), which is not capable of speciating hydrocarbons. The Multigas, on the other hand, does speciate hydrocarbons, and the total hydrocarbons are summed from the species indicated in

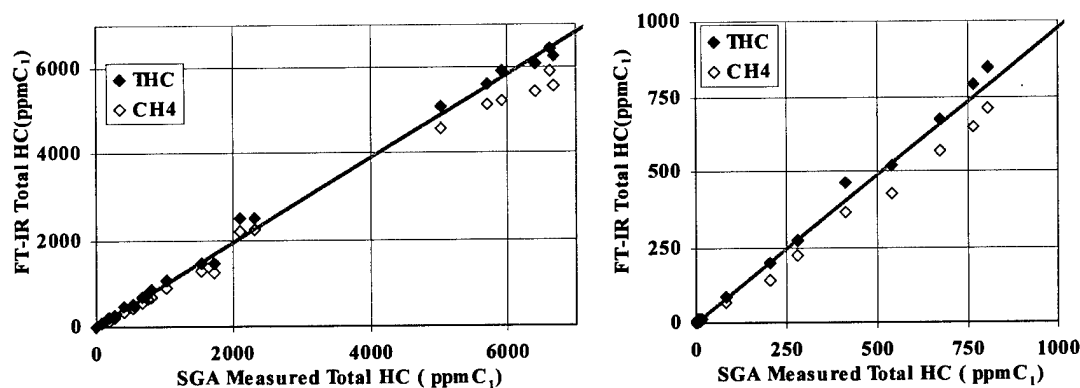


Figure 7. Correlation plot for Total Hydrocarbons. The graph on the left covers the entire dynamic range of the data, while the right expands the data from 0-1000 ppm. The total Hydrocarbons measured with the Multigas is calculated using the species listed in **Table III**. The best linear fit shown has a slope of 0.975 and zero offset. Also plotted is the correlation between Multigas measured methane and the THC's. See text for details.

Species	Fraction of total C ₁ (%)
Methane	80-90%
Formaldehyde	7-8%
Ethylene	8-9%
Methanol	<1%
Propylene	<1%
Formic acid	<1%

Table III. Hydrocarbons detected in the turbine combustor exhaust, and the relative contribution of each. These contributions are normalized to the FID analyzer results, and have been corrected by the number of carbon atoms in each molecule.

Table III. This table lists all the hydrocarbon compounds detected in the turbine exhaust, and includes the approximate percent contribution to the total hydrocarbon concentration as reported by the FID. Since the FID results are normalized to C₁ (i.e. equivalent methane), the relative contribution for each compound in the table has also been normalized to C₁. As seen in this table, as well as in **Figure 7**, the Multigas results are typically within about 2.5% of the FID results, which is well within the experimental uncertainty of the measurements.

Measured and Calculated Water Comparision

In addition to the species reported in **Figures 3-7** and **Table III**, water was measured directly using the Multigas. The water concentrations measured had an excellent linear correlation with the calculated water concentration reported by AEDC, but was systematically low by 5%. Since the fuel C-H ratio was

measured on a regular basis, the water concentration can be calculated with good accuracy based on the test rig stoichiometry, inlet humidity, and known fuel C-H ratio. This approach is most accurate when rig is operating at high combustion efficiency (i.e. no unburned hydrocarbons or CO). The approximately 10% error in the C-H ratio calculated using the Multigas data has led to the identification of a systematic error in the water references used. This error arose out of the difficulty in generating precise water references at the levels necessary for combustion monitoring (up to 13% water in the present study, but much higher for other programs). For this program, a heated water bubbler was used to generate the references, and systematic errors in the bubbler temperature by as little as 1°C can cause a 5% error due to the strong temperature dependence of water's vapor pressure.⁴ Based on the precision that the water concentration can be calculated with in the test rig, all the water reference concentrations used were re-scaled by a constant factor of 1.05.

CONCLUSIONS

The FT-IR based Multigas analyzer has been used to successfully monitor the exhaust composition from a test turbine combustor. The instrument was able to demonstrate its ability to not only measure the species traditionally measured at the facility (CO₂, CO, NO, NO_x, and total hydrocarbons), but also to detect and measure many other species, including water, formaldehyde, ethylene, propylene, methanol, formic acid, and nitrous acid. The only traditional analyzer that the Multigas is unable to compete with is the oxygen analyzer, since molecular oxygen is not infrared active.

The side by side comparison of the single gas analyzers currently employed by AEDC and the MG-2010 Multigas analyzer showed excellent agreement, indicating the utility of the Multigas as an extremely versatile analyzer. This versatility coupled with the analyzer's relatively small footprint, ease of use, minimal daily calibration, and low maintenance, make the Multigas an ideal instrument for both laboratory and field measurements of turbine engine exhaust composition.

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